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STUDIES ON METHOD FOR AQUEOUS EXTRACTION OF SOYBEAN OIL

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Abstract: Water extraction of soybean oil was studied to find the optimal conditions for recovery of oil pre-enriched protein and for aqueous extraction of soybean oil. Orthogonal tests were employed in the procedures of oil pre-enrichment and aqueous extraction. Soybeans were crushed to pass a 40 mesh sieve, soaked under the optimum conditions (solid/water = 1/5(w/v), 40 ℃, pH 10, 3 h) and water-ground to 100 mesh, stirred in 65 ℃ water for 20 min, and centrifuged at 1400 g to separate oil pre-enriched protein. The protein yield was 17.8 g from 100 g soybeans, which contained 62.8% oil. The oil yield was 69.0%. Optimum conditions for the aqueous extraction procedure were: solid-to-water ratio 1:2, pH 9.0, time 30 min, stirring in boiling water bath, stationary time 10 min, centrifuge at 3600 g for 10 min. Experimental values showed that the oil yield after aqueous extraction from oil pre-enriched protein reached 88.3%, so the total oil extraction rate was 60.8%.

Key words: soybean oil, aqueous extraction, pre-enrichment
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INTRODUCTION

Vegetable oil can be extracted with water. The method's advantages are retention of the original properties of proteins and low processing cost. Sugarman (1956) first described the technology of aqueous oil extraction 1956. In 1972, Rhee (1972) improved the oil yield of the technology. In more than 20 years since then, the method has been successfully applied for processing oil from peanut seed, rape seed and cotton seed. However, if applied to raw oil material (such as soybean) with low levels of oil, the yield was very low (Lawhon et al., 1981). In order to raise the yield, Adler-Nissen (1978) used a protease to separate the proteins and oil. In this technology, the proteins were degraded by an enzyme to release the oil so that the oil yield was improved to nearly 60%. In 1994, Wang and coworkers (Wang et al., 1994) isolated oil and protein by degrading the proteins by using an edible protease to solubilize them and obtained 66% oil yield. But the cost was too high for the method to be industrialized because a great deal of edible protease (more than 7000 unit/g soybean) was used. In the present research, the oil was pre-enriched in the aqueous mixture containing oil and proteins. Soybean proteins containing 62.8% oil were separated from the mixture. Then the oil was extracted with alkaline boiling water from the proteins, centrifuged, and 60.8% of oil yield was obtained. This technology has good application potential because of its low cost. The difficulty of aqueous extraction of soybean oil has been solved.

MATERIALS AND METHODS

1. Materials

Soybeans produced in Henan Province, China, were purchased in a local market. The beans contained 12.3% moisture, 17.1% oil and 39.2% protein.

2. Soybean oil pre-enriched with water

The soybeans were crushed and soaked in warm water with controlled solid-to-liquid ratio, then was water-ground to pass a 100 mesh sieve. The pH of the liquid was adjusted to pH 10 in a stirred vessel. Temperature was regulated by a water bath. The oil-enriched protein was separated by centrifugation.

3. Aqueous extraction of soybean oil

The oil in separation protein solid residue was then extracted with boiling alkaline water accompanied by stirring, allowed to settle and centrifuged to obtain soybean oil.

4. Analytical methods

Moisture and crude protein were determined according to standard methods (AOAC, 1990). Oil was measured by the refraction method (WLII, 1983).

RESULTS AND DISCUSSION

1. Soybean oil pre-enriched in protein with water

Soybeans consist of about 17% oil and 39% protein. The crushed soybeans were soaked in warm water because the more oil protein emulsion that was extracted, the easier it was to increase the yield of soybean oil. The degree of crushing, the soaking ratio of solids to liquid, the soaking temperature and soaking time were varied to determine the optimum extraction of oil-protein emulsion from soybeans water-ground to pass 100-mesh (Figs 1 – 4).

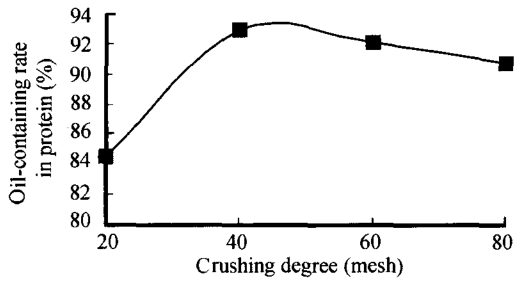

Fig.1 Effect of elementary crushing degree on the extraction by water (solid-to-liquid ratio 1:7, soaked at 50 ℃ for 4 h)

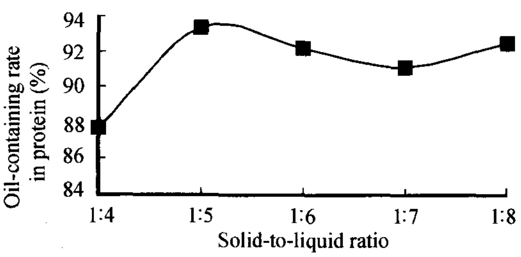

Fig.2 Effect of solid-to-liquid ratio on the extraction by water (40 mesh, soaked at 50 ℃ for 4 h)

The preferred parameters were: crushing degree 40 mesh, ratio of solid to liquid 1:5, water temperature 40 ℃, and an extraction time of 3 h, which resulted in a oil yield of 93.5% of oil mixture soybean protein.

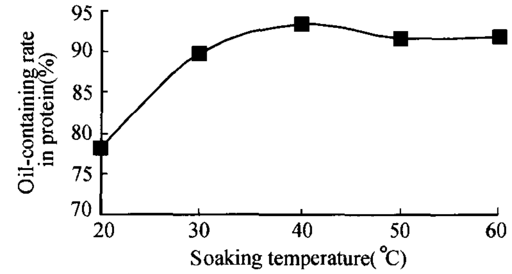

Fig.3 Effect of soaking temperature on the extraction by water (40 mesh, solid-to-liquid ratio 1:7, soaked for 4 h)

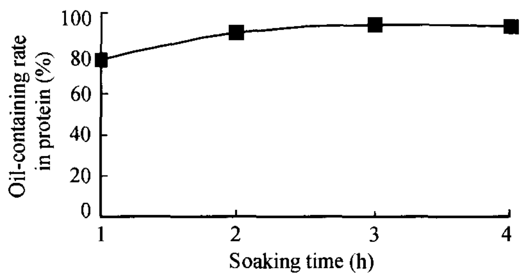

Fig.4 Effect of soaking time on the extraction by water (40 mesh, solid-to-liquid ratio 1:5, soaked at 40 ℃)

Crushed and soaked soybeans were processed in several preliminary tests to determine the effects of water grinding, pH of the suspension, stirring time and temperature. Soybeans were water-ground to pass 120 mesh, the pH was set to 9.0, stirred at 45 ℃ for 30 min, and centrifuged at 1400g for 15 min. The oil content rate in dry basis was 45.2% and the oil yield was 49.1%. We expected that soybeans will release part of the oil after soaking and grinding in alkaline solution. The released oil was adsorbed by protein, which precipitated during centrifugation. Due to the slightly alkaline medium, the amount of precipitated protein was reduced.

After the slurry was centrifuged at 1400 g for 15 min, orthogonal test was applied to determine optimum conditions. The factors and levels of the orthogonal test selected are shown in Table 1.

Table 2 shows the results of the orthogonal test. The amount of oil contained in enriched solid and the extraction rate of soybean oil were determined by range analysis. pH of the medium was the most significant factor, with stronger alkalinity of solution being better. Because there were no great changes as pH was increased from 9 to 10, the pH was set to 10, and higher pH values were not tested. The effect of 20 min stirring time period was similar to that of 60 min, and both were more efficient than 40 min. We expect that the relationship between oil release and adsorption is that release occurs first, then adsorption becomes dominant, and finally, release takes priority again, and so on, to create a reaction equilibrium. So, we fixed the stirring time at 20 min. High temperature facilitates oil release and facilitates oil enrichment. When temperature was increased from 50 ℃ to 65 ℃, the effects on oil yield and oil extraction were not significant (1.5%). Because high temperature will denature protein, the temperature was set to 65 ℃ and was not increased any further. The degree of water grinding was the least critical. As the degree was increased from 100 mesh to 120 mesh both the oil yield and oil extraction rate decline significantly.

Table 1 Factor and level design of orthogonal test*

Factor	Water grinding degree *A* (mesh)	pH of medium *B*	Temperature *C* (℃)	Time *D* (min)
Level 1	140	8	35	20
Level 2	120	9	50	40
Level 3	100	10	65	60

* Test amount: 100 g

Table 2 Results of the orthogonal test of soybean oil enrichment with water

Test No.	*A*	*B*	*C*	*D*	Solid dry weight (g)	Oil containing rate (dry basis) (%)	Oil yield (%)
1	1	1	1	1	17.0	36.0	35.7
2	1	2	2	2	18.1	46.1	48.8
3	1	3	3	3	18.9	59.6	65.7
4	2	2	3	1	18.6	57.7	62.6
5	2	3	1	2	17.4	40.7	41.3
6	2	1	2	3	17.9	42.5	44.4
7	3	3	2	1	19.1	62.9	70.1
8	3	1	3	2	17.7	38.3	39.5
9	3	2	1	3	18.5	56.1	60.5
R1*	47.2	38.9	44.3	52.2			
R2*	47.0	53.3	50.5	41.7			
R3*	52.4	54.4	51.9	52.7			
R*	5.4	15.5	7.6	11.0			
R1**	50.1	39.9	45.8	56.1			
R2**	49.4	57.3	54.4	43.2			
R3**	56.7	59.0	55.9	56.9			
R**	7.3	19.1	10.1	13.7			

Factor order: $B > D > C > A$
* Oil containing rate (dry basis)
** Oil yield

Table 3 shows that 100 mesh was the optimum. We believe that the particle fineness degree was closely related with oil release and absorption, and that 100 mesh was beneficial to oil adsorption during the release and precipitation stage.

Under the above optimum conditions, three more experiments (each with 500 g of soybeans) were conducted to achieve the average results: enriched solid weighs 17.8g/100 g soybean, oil content of 62.8% (dry basis), and oil extraction of 69.0%.

Table 3 Effect of water grinding on the enrichment of soybean oil*

Water grinding (mesh)	140	120	100	80
Oil yield (%)	49.8	60.1	69.4	58.7

* Test amount 100 g, pH 10, at 65 ℃ for 20 min, 1400 g centrifugation

2. Aqueous extraction of soybean oil

Based on the traditional aqueous extraction method, all tests began with oil-enriched soybean protein, centrifuged for 10 min. First tested was the centrifugation rate for aqueous extraction. When the emulsion was added to boiling water, with regulated temperature for 40 min, and was centrifuged at 4000 g, the upper layer was the oil; when centrifuged at 2200 g, the upper layer had oil beads and was mixed with protein, indicating that the emulsion was broken more extensively by centrifuging at higher rate. Then effect of the pH value of water was determined. When wet protein was added to twice its volume of boiling water, the mixture was maintained at natural pH, and constant temperature for 40 min, an upper layer of oil formed; when pH was 9.0, the upper layer had more oil. So, slightly alkaline water solution was better. The final test was on the stirring. When the wet protein plus twice its volume of boiling water was stirred for 30 min, then left stationary for 10 min, the upper oil layer was thick. Without stirring, the upper oil layer was thin. It showed that stirring can help to increase the oil yield during the process. Thus, an orthogonal test with the factors and levels shown in Table 4 was arranged to determine the optimum conditions. In the experiments, the stationary time period, after stirring was set at 10 min, and the oil containing protein aqueous solution was slightly cold, centrifugation was controlled at 1400 g for 10 min. Oil-containing protein was separated from the upper layer, and the extraction rate calculated on a dry basis. Results are shown in Table 5.

Table 4 Factors and levels in orthogonal test of aqueous extraction*

Factor	Centrifugation rate	Water pH	Water amount-to-oil containing protein ratio	Stirring time
	A (g)	*B*	*C*	*D* (min)
Level 1	4200	7.0	1.5:1	20
Level 2	3600	8.0	2.0:1	30
Level 3	3000	9.0	2.5:1	40

* Test amount: 100 g

Table 5 Results of the orthogonal test of aqueous extraction

Test No.	*A*	*B*	*C*	*D*	Oil yield (%)
1	1	1	1	1	73.1
2	1	2	2	2	82.9
3	1	3	3	3	85.6
4	2	1	2	3	80.2
5	2	2	3	1	77.8
6	2	3	1	2	88.3
7	3	1	3	2	78.4
8	3	2	1	3	83.0
9	3	3	2	1	83.7
R1	80.5	77.2	81.5	78.2	
R2	82.1	81.2	82.3	83.2	
R3	81.7	85.9	80.6	82.9	
R	1.6	8.7	1.7	5.0	

Table 5 indicates that the pH of the extraction water was the most significant factor. Slightly alkaline solution was favorable for the extraction. Because of the denaturalization of protein in alkaline solution, the experiment pH was set at 9.0. Stirring in boiling water for 30 min was best because longer or shorter time periods do not increase the oil yield. The volume of extraction water was twice that of the oil-containing protein. The centrifugation rate was 3600 g. After the above conditions were determined, tests on the resting period after stirring (Table 6) and the centrifugation time (Table 7) were done for the sake of industrial application.

Table 6 Effect of stationary time after stirring on the extraction*

Stationary time (min)	5	10	15	20	25
Oil yield (%)	86.7	88.2	88.0	87.1	85.8

* Centrifugation for 10 min

Table 7 Effect of centrifugation time on the extraction*

Centrifugation time (min)	5	10	15	20	25
Oil yield (%)	79.5	88.1	88.6	88.2	88.4

* Stationary for 10 min

Table 6 shows a 10-min stationary time was best for the oil yield; longer time enhanced the adsorption of protein, and shorter time adversely affected the congregation of the oil phase, dispersed in solution by stirring. Table 7 shows that centrifuging for 10 min was enough for the separation.

To verify the predicted values, a series of aqueous extraction experiments under the optimum conditions (centrifugation rate 3600 g, water pH 9.0, solid-to-water ratio 1:2, stirring time 30 min, stationary time 10 min and centrifugation for 10 min) was carried out to obtain average oil yield of 88.3% (dry basis). When the water pH was changed to 10.0, the oil yield was 89.1% (dry basis). For soybean protein for food , pH was set at 9.0.

CONCLUSIONS

When soybeans are processed by the pre-enrichment and aqueous extraction procedures, the average yield of pre-enriched soybean oil is 69.0%; the average yield of oil out put by aqueous extraction from oil-enriched protein can reach to 88.3%, the total oil yield to 60.8%. The moisture of the extracted oil was 0.13%; the oil appeared slightly colored and is edible. The techniques of oil-enrichment in water phase and aqueous extraction of soybean oil are feasible. In the complete process, enrichment serves as a control step.

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